



**ALLIANCE**  
Technologies Corporation

*Ciba Geigy*  
**received**  
02 aug 91 MH

TRC/T6  
9104018-IA

REC'D  
8-5-91  
FB.

July 24, 1991

Dennis Gagne  
Regional Sample Control Custodian  
U.S. Environmental Protection Agency  
90 Canal Street  
Boston, MA 02114

Re: 68-W9-0003  
Work Assignment R01005  
Case 16259, SDG MAT367  
Skinner and Sherman  
Ciba-Geigy  
Metals: 6/Water  
Cyanide: 6/Water

Dear Mr. Gagne:

The following is a Data Validation Report for CLP case 16259, which was generated by QuantaLex Inc., Alliance's Data Validation Sub-Contractor for this work assignment. The inorganic analytical data for this case contained low level water samples which were collected by Alliance Technologies Corporation at the Ciba-Geigy Site and analyzed by Skinner and Sherman.

If you have any questions, please feel free to contact me at (508) 970-5600 X 4201.

Sincerely,

Cynthia S. Fortin  
Data Validation Coordinator

encl.

cc: Deborah Szaro/Moira Lataille, Region I TPO



SEMS DocID

666774

June 19, 1991

Ms. Joanna Hall  
Alliance Technologies Corp.  
Boott Mills South, Foot of John Street  
Lowell, MA 01852

Re: Case 16259, SDG MAT367  
Skinner & Sherman  
Metals: 6/Water  
Cyanide: 6/Water

Dear Ms. Stallings:

A validation was performed on the analytical data from six water samples which were collected by Alliance Technologies Corp. and submitted to Skinner & Sherman for Inorganic analysis. The data were evaluated based on the following parameters:

- \* ■ Data completeness
- \* ■ Holding times
- Calibration verification
- Laboratory and field blank analyses
- \* ■ ICP interference check sample results
- Matrix spike recoveries
- Laboratory and field duplicates
- \* ■ Laboratory control sample results
- Furnace atomic absorption results
- Serial dilution results
- \* ■ Detection limit results
- \* ■ Sample results

\* - All criteria were met for this parameter

Table 1 summarizes the validation recommendation which were based on the following information.

Calibration Verification

<u>Element</u>	<u>CRI</u> <u>True Value</u>	<u>Found</u>	<u>%R</u>
Cr	20.0	15.4	77.1
Cu	50.0	61.6	61.6
Zn	40.0	51.5	51.5

All positive values less than 3X CRDL are estimated (J).

The Selenium initial calibration correlation coefficient was less than 0.995. All non-detects are estimated (UJ).

Blanks

<u>Element</u>	<u>Maximum Conc./Units</u>	<u>Action Level</u>
Ba	10.4 ug/L	52 ug/L
Co	5.0 ug /L	25 ug/L
Cu	19.1 ug/L	95.5 ug/L
Zn	11.7 ug/L	58.5 ug/L

Value > IDL, < CRDL, and < Action Level = Report value estimated (UJ).

Value > IDL, > CRDL, and < Action Level = Report value U.

Value > IDL and > Action Level = Report value unqualified.

Value < IDL and blank < -IDL = Raise detection limits to CRDL and Report value UJ.

Matrix Spike

<u>Analyte</u>	<u>SSR</u>	<u>SR</u>	<u>S</u>	<u>%R</u>
Se	5.16 ug/L	20.0 U ug/L	10.0 ug/L	51.6

Positive results are estimated (J) and non-detects are estimated (UJ) when spike recovery is between 30%-74%.

Field Duplicates

<u>Analyte</u>	<u>Sample Result</u>	<u>Duplicate Result</u>	<u>RPD</u>
Al	3680 ug/L	5340 ug/L	36.85.5

Positive results are estimated (J).

Furnace Atomic Absorption Results

<u>IIR #</u>	<u>Analyte</u>	<u>Sample Result/ Units</u>	<u>Analytical Spike Result/Units</u>	<u>% Recovery</u>
MAT368	Se	4.0 U ug/L	6.99 ug/L	70%
MAT369	Se	4.0 U ug/L	8.30 ug/L	83%
MAT370	Se	4.0 U ug/L	5.23 ug/L	52%
MAT371	Se	4.0 U ug/L	7.86 ug/L	79%
MAT367	Tl	20.0 U ug/L	13.8 ug/L	69%
MAT368	Tl	3.0 U ug/L	8.95 ug/L	45%
MAT370	Tl	3.0 U ug/L	15.2 ug/L	76%
MAT371	Tl	3.0 U ug/L	14.9 ug/L	74%

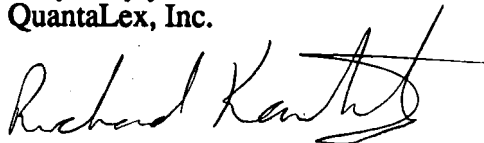
Non-detect sample values are estimated (UJ) if spike recovery < 85%.

ICP Serial Dilution Results

<u>Analyte</u>	<u>Sample Result</u>	<u>Serial Dilution</u>	<u>%D</u>
Cr	162.34 ug/L	137.15 ug/L	15.5

All positive values are estimated (J).

Very truly yours,  
QuantaLex, Inc.



Richard Kantrowitz  
Associate Consultant

cc: Keith Wegner, President

**SKINNER & SHERMAN  
CASE 16259**

**TABLE I - RECOMMENDATIONS SUMMARY**

Aluminum	J7	Magnesium	A
Antimony	A	Manganese	A
Arsenic	A	Mercury	A
Barium	A <sup>1</sup>	Nickel	A
Beryllium	A	Potassium	A
Cadmium	A	Selenium	J1,J4,J6
Calcium	A	Silver	A
Cobalt	J3	Sodium	A
Chromium	J2,J5	Thallium	J4
Copper	A1,J3,J5	Vanadium	A
Iron	A	Zinc	A1,J5
Lead	A	Cyanide	A

- A - Accept all data.
- A<sup>1</sup> - Accept data, raise the sample detection limit(s) due to blank contamination.
- J1 - Estimate (UJ) non-detects due to poor pre-digestion matrix spike recovery.
- J2 - Estimate (J) positive values due to poor serial dilution recovery.
- J3 - Estimate (UJ) positive values due to blank contamination and values < CRDL.
- J4 - Estimate (UJ) non-detects due to poor analytical spike recovery.
- J5 - Estimate (J) positive values less than 3X CRDL due to poor CRDL check standard recovery.
- J6 - Estimate (UJ) non-detects due to initial calibration correlation coefficient < 0.995.
- J7 - Estimate (J) positive values due to poor duplicate precision.

# INORGANIC REGIONAL DATA ASSESSMENT

Region 1

CASE NO. 16259  
 LABORATORY Skinner & Sherman  
 SDG # MAT367  
 SOW # 7/88  
 DPO: ACTION \_\_\_\_\_ FYI \_\_\_\_\_

SITE Ciba-Geigy  
 NO. OF SAMPLES/             
 MATRIX 6/Water  
 REVIEWER (IF NOT ESD) QuantaLex, Inc.  
 REVIEWER'S NAME Richard Kantrowitz  
 COMPLETION DATE June 19, 1991

## Data Assessment Summary

	ICP	AA	Hg	Cyanide
1. Holding Times	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
2. Calibrations	<u>O</u>	<u>M</u>	<u>O</u>	<u>O</u>
3. Blanks	<u>M</u>	<u>O</u>	<u>O</u>	<u>O</u>
4. ICS	<u>O</u>	<u>N/A</u>	<u>N/A</u>	<u>N/A</u>
5. LCS	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
6. Duplicate Analysis	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
7. Matrix Spike	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
8. MSA	<u>N/A</u>	<u>O</u>	<u>N/A</u>	<u>N/A</u>
9. Serial Dilution	<u>O</u>	<u>N/A</u>	<u>N/A</u>	<u>N/A</u>
10. Sample Verification	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
11. Other QC	<u>O</u>	<u>O</u>	<u>O</u>	<u>O</u>
12. Overall Assessment	<u>M</u>	<u>M</u>	<u>O</u>	<u>O</u>

O = Data had no problems/or qualified due to minor problems.

M = Data qualified due to major problems.

Z = Data unacceptable.

X = Problems, but do not affect data.

N/A = Not applicable.

ACTION ITEMS: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

AREAS OF CONCERN: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

NOTABLE PERFORMANCE: \_\_\_\_\_  
 \_\_\_\_\_  
 \_\_\_\_\_

REGION I  
Data Review Worksheets

Site Name Ciba-Geigy  
Reference Number \_\_\_\_\_

REGION I REVIEW OF INORGANIC  
CONTRACT LABORATORY DATA PACKAGE

The hardcopied Skinner & Sherman data package received at Region I has been reviewed and the quality assurance and performance data summarized. The data reviewed included:

Case No.	<u>16259</u>	SAS No.	_____	Sampling Date(s):	<u>04/18/91</u>
SDG No.	<u>MAT367</u>	Matrix	<u>Water</u>	Shipping Date(s):	<u>04/19/91</u>
No. of Samples	<u>6</u>			Date Rec'd by Lab:	<u>04/20/91</u>

Traffic Report Nos.: MAT367, MAT368, MAT369, MAT370, MAT371, MAT372

Trip Blank No.: MAT372

Equipment Blank No.: \_\_\_\_\_

Field Dup. Nos.: MAT367, MAT368

SOW No. 7/88 requires that specific analytical work be done and that associated reports be provided by the laboratory to the Regions, EMSL-LV, and SMO. The general criteria used to determine the performance were based on an examination of:

- |                                  |                               |
|----------------------------------|-------------------------------|
| - Data Completeness              | - Field Duplicates            |
| - Holding Times                  | - Lab Control Sample Results  |
| - Calibrations                   | - Furnace AA Results          |
| - Blanks                         | - ICP Serial Dilution Results |
| - ICP Interference Check Results | - Detection Limit Results     |
| - Matrix Spike Recoveries        | - Sample Quantitation         |
| - Laboratory Duplicates          |                               |

Overall comments: Data acceptable with qualifications.

Definitions of Qualifiers:

- A - Acceptable data.  
J - Approximate data due to quality control criteria.  
R - Reject data due to quality control criteria.  
U - Compound not detected.

Reviewer: Richard Kontny

Date: 6/19/91

## REGION I

### Data Review Worksheets

## I. DATA COMPLETENESS

### MISSING INFORMATION

DATE LAB CONTACTED

DATE RECEIVED

None



**REGION I**  
**Data Review Worksheets**

**II. HOLDING TIMES**

Complete table for all samples and circle the fractions which are not within criteria.

SAMPLE ID	DATE SAMPLED	Hg DATE ANAL	CYANIDE DATE ANAL	OTHERS DATE ANAL	pH	ACTION
MAT367	04/18/91	05/09/91	05/02/91	05/09/91		None
MAT368	04/18/91	05/09/91	05/02/91	05/09/91		None
MAT369	04/18/91	05/09/91	05/02/91	05/09/91		None
MAT370	04/18/91	05/09/91	05/02/91	05/09/91		None
MAT371	04/18/91	05/09/91	05/02/91	05/09/91		None
MAT372	04/18/91	05/09/91	05/02/91	05/09/91		None

**METALS** - 180 days from sample collection  
**MERCURY** - 28 days from sample collection  
**CYANIDE** - 14 days from sample collection

**ACTION:**

1. If holding times are exceeded, all positive results are estimated (J) and non-detects are estimated (UJ).
2. If holding times are grossly exceeded, the reviewer may determine that non-detects are unusable (R).

REGION I  
Data Review Worksheets

**III A. INSTRUMENT CALIBRATION (Section 1)**

**1. Recovery Criteria**

List the analytes which did not meet the percent recovery (%R) criteria for Initial or Continuing Calibration.

<u>DATE</u>	<u>ICV/CCV#</u>	<u>ANALYTE</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
<u>None</u>					

**ACTIONS:**

If any analyte does not meet the %R criteria, follow the actions stated below:

For Positive Results:

	<u>Accept</u>	<u>Estimate (J)</u>	<u>Reject (R)</u>
Metals	90-110%R	75-89%R, 111-125%R	<75%R, >125%R
Mercury	80-120%R	65-79%R, 121-135%R	<65%R, >135%R
Cyanide	85-115%R	70-84%R, 116-130%R	<70%R, >130%R

For Non-detected Results:

	<u>Accept</u>	<u>Estimate (UJ)</u>	<u>Reject (R)</u>
Metals	90-125%R	75-89%R	<75%R, >125%R
Mercury	80-135%R	65-79%R	<65%R, >135%R
Cyanide	85-130%R	70-84%R	<70%R, >130%R

### III B. INSTRUMENT CALIBRATION (Section 2)

#### 2. Analytical Sequence

- |    |   |     |
|----|---|-----|
| A. | Did the laboratory use the proper number of standards for calibration as described in the SOW?  | Yes |
| B. | Were calibrations performed at the beginning of each analysis?  | Yes |
| C. | Were calibration standards analyzed at the beginning of sample analysis and at a minimum frequency of ten percent or every two hours during analysis, whichever is more frequent? | Yes |
| D. | Were the correlation coefficients for the calibration curves for AA, Hg, and CN $\geq 0.995$ ?  | No  |
| E. | Was a standard at 2xCRDL analyzed for all ICP analyses?   | Yes |

If no,  
the data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

Se (run 1, 5/8/91) initial calibration correlation coefficient was equal to 0.9929.

Se (run 2, 5/9/91) initial calibration correlation coefficient was equal to 0.9858.

The CRDL check sample recovery for Cr was 77.1%.

The CRDL check sample recovery for Cu was 123.2%.

The CRDL check sample recovery for Zn was 128.8%.

REGION I  
Data Review Worksheets

IV A. BLANK ANALYSIS RESULTS (Sections 1-3)

List the blank contamination in Sections 1 and 2 below. A separate worksheet should be used for soil and water blanks.

1. Laboratory Blanks

Matrix: Water

<u>DATE</u>	<u>ICB/CCB#</u>	<u>PREP BL</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
05/09/91	CCB4		Al	41.8 ug/L
05/09/91	CCB4		Sb	23.5 ug/L
05/09/91	CCB1		Ba	10.4 ug/L
05/09/91	CCB4		Cd	3.8 ug/L
05/09/91	CCB1		Ca	32.8 ug/L
05/09/91		PB	Ca	-17.2 ug/L
05/09/91	CCB4		Co	5.0 ug/L
05/09/91	CCB4		Cu	19.1 ug/L
05/09/91	CCB1		Fe	55.0 ug/L
05/09/91	CCB1		Mn	2.8 ug/L
05/09/91	CCB4		K	136 ug/L
05/09/91		PB	Na	56.3 ug/L
05/09/91	ICB		Zn	11.7 ug/L
05/09/91	CCB3		Pb	-1.4 ug/L

2. Equipment/Trip Blanks

<u>DATE</u>	<u>EQUIP BL#</u>	<u>ANALYTE</u>	<u>CONC./UNITS</u>
05/09/91	MAT372	Al	20.5 ug/L
05/09/91	MAT372	Cu	9.4 ug/L
05/09/91	MAT372	Pb	1.1 ug/L
05/09/91	MAT372	Na	128 ug/L
05/09/91	MAT372	Zn	10.3 ug/L

3. Frequency Requirements

- A. Was a preparation blank analyzed for each matrix, for every 20 samples, and for each digestion batch? Yes
- B. Was a calibration blank run every 10 samples or every 2 hours, whichever is more frequent? Yes

If no,

the data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

#### IV B. BLANK ANALYSIS RESULTS (Section 4)

##### 4. Blank Actions

The Action Levels for any analyte is equal to five times the highest concentration of that element's contamination in any blank. The action level for samples which have been concentrated or diluted should be multiplied by the concentration/dilution factor. No positive sample result should be reported unless the concentration of the analyte in the sample exceeds the Action Level (AL). Specific actions are as follows:

1. When the concentration is greater than the IDL, but less than the Action Level, report the sample concentration detected with a U.
2. When the sample concentration is greater than the Action Level, report the sample concentration unqualified.

MATRIX: Water

<u>ELEMENT</u>	<u>MAX. CONC./ UNITS</u>	<u>AL/ UNITS</u>
<u>Ba</u>	<u>10.4 ug/L</u>	<u>52.0 ug/L</u>
<u>Co</u>	<u>5.0 ug/L</u>	<u>25.0 ug/L</u>
<u>Cu</u>	<u>19.1 ug/L</u>	<u>95.5 ug/L</u>
<u>Zn</u>	<u>11.7 ug/L</u>	<u>58.5 ug/L</u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>

MATRIX:  

<u>ELEMENT</u>	<u>MAX. CONC./ UNITS</u>	<u>AL/ UNITS</u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>
<u> </u>	<u> </u>	<u> </u>

NOTE: Blanks analyzed during a soil case must be converted to mg/kg in order to compare them with the sample results.

$$\text{Conc. in ug/L} \times \frac{\text{Volume diluted to (200 ml)}}{\text{Weight digested (1 gram)}} \times \frac{1\text{L}}{1000\text{ml}} \times \frac{1000\text{ gm}}{1\text{ kg}} \times \frac{1\text{ mg}}{1000\text{ug}} = \text{mg/kg}$$

Multiplying this result by 5 to arrive at the action level gives a final result in mg/kg which can then be compared to sample results.

**V A. ICP INTERFERENCE CHECK SAMPLE (Sections 1 and 2)**

**1. Recovery Criteria**

List any elements in the ICS AB solution which did not meet the criteria for %R.

<u>DATE</u>	<u>ELEMENT</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
None				

**ACTIONS:**

If an element does not meet the %R criteria, follow the actions stated below:

	<u>Percent Recovery</u>		
	<u>&lt;50%</u>	<u>50%-79%</u>	<u>&gt;120%</u>
Positive sample results	R	J	J
Non-detected results	R	UJ	A

**2. Frequency Requirements**

Were interference QC samples run at the beginning and end of each sample analysis run or a minimum of twice per 8 hour working shift, whichever is more frequent?

Yes

If no,

the data may be affected. Use professional judgement to determine the severity of the effect and qualify the data accordingly. Discuss any actions below and list the samples affected.

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REGION I  
Data Review Worksheets

**V B. ICP INTERFERENCE CHECK SAMPLE (Section 3)**

3. Report the concentration of any elements detected in the ICS A solution > 2xIDL that should not be present.

ELEMENT	CONC. DETECTED IN THE ICS	CONC. OF INTERFERENTS IN THE ICS			
		AL	CA	FE	MG
Ba	12	481000	448250	182650	450900
Cd	6	481000	448250	182650	450900
Cu	14	481000	448250	182650	450900
Ni	11	481000	448250	182650	450900
Ag	-15	481000	448250	182650	450900
Na	1732	481000	448250	182650	450900
Zn	216	481000	448250	182650	450900

Estimate the concentration produced by the interfering element in all affected samples. See guidelines for examples. List the samples affected by interferences below:

SAMPLE AFFECTED	ELEMENT AFFECTED	SAMPLE CONC. (ug/L)	SAMPLE INTERFERENT CONC.				ESTIMATED INTERF. (ug/L)
			AL	CA	FE	MG	
None							

**ACTIONS:**

1. In general, the sample data can be accepted without qualification if the sample concentrations of Al, Ca, Fe, and Mg are less than 50% of their respective levels in the ICS solution.
2. Estimate (J) positive results for affected elements for samples with levels of interferences 50% or more of that in the ICS solution.
3. Reject (R) positive results if the reported concentration is due entirely to the interfering element.
4. Estimate (UJ) non-detected results for which false negatives are suspect.

Give explanations for any actions taken below:

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REGION I  
Data Review Worksheets

VI. MATRIX SPIKE

TR # MAT371S

MATRIX: Water

1. Recovery Criteria

List the percent recoveries for analytes which did not meet the required criteria.

SSR = Spikes sample result

SR = Sample result

S = Amount of spike added

<u>ANALYTE</u>	<u>SSR</u>	<u>SR</u>	<u>S</u>	<u>%R</u>	<u>ACTION</u>
Se	5.16 ug/L	20.0 U ug/L	10.0 ug/L	51.6	J/UJ

Matrix Spike Actions apply to all samples of the same matrix.

ACTIONS:

- If the sample concentration exceeds the spike concentration by a factor of 4 or more, no action is taken.
- If any analyte does not meet the %R criteria, follow the actions stated below:

	<u>Percent Recovery</u>		
	<u>&lt;30%</u>	<u>30%-74%</u>	<u>&gt;125%</u>
Positive sample results	J	J	J
Non-detected results	R	UJ	A

2. Frequency Criteria

- Was a matrix spike prepared at the required frequency? Yes
- Was a post digestion spike analyzed for elements that did not meet required criteria for matrix spike recovery? Yes

A separate worksheet should be used for each matrix spike pair.



## VII. LABORATORY DUPLICATES

List the concentrations of any analyte not meeting the criteria for duplicate precision. For soil duplicates, calculate the CRDL in mg/kg using the sample weight, volume, and percent solids data for the sample. Indicate what criteria was used to evaluate precision by circling either the RPD or CRDL for each element.

MATRIX: Soil

<u>Element</u>	<u>CRDL</u>		<u>Sample #</u> <u>MAT371</u>	<u>Duplicate #</u> <u>MAT371D</u>	<u>RPD</u>	<u>Action</u>
	<u>water</u> ug/L	<u>soil</u> mg/kg				
Aluminum	200	None				
Antimony	60					
Arsenic	10					
Barium	200					
Beryllium	5					
Cadmium	5					
Calcium	5000					
Chromium	10					
Cobalt	50					
Copper	25					
Iron	100					
Lead	3					
Magnesium	5000					
Manganese	15					
Mercury	0.2					
Nickel	40					
Potassium	5000					
Selenium	5					
Silver	10					
Sodium	5000					
Thallium	10					
Vanadium	50					
Zinc	20					
Cyanide	10					

Laboratory Duplicate Actions should be applied to all other samples of the same matrix type.

### ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >20% for waters and >35% for soils.
2. If sample results are less than 5x the CRDL, estimate (J) positive results for elements whose absolute difference is >CRDL (2xCRDL for soils). If both samples are non-detected, the RPD is not calculated (NC).

## VIII. FIELD DUPLICATES

List the concentrations of all analytes in the field duplicate pair. For soil duplicates, calculate the CRDL in mg/kg using the sample weight, volume, and percent solids data for the sample. Indicate what criteria was used to evaluate the precision by circling either the RPD or CRDL for each element.

MATRIX: Soil

Element	CRDL		Sample # MAT367	Duplicate # MAT368	RPD	Action
	water ug/L	soil mg/kg				
Aluminum	200		3680	5340	36.8	J/A
Antimony	60		17.0 U	17.0 U	NC	None
Arsenic	10		19.7	13.3	38.8	None
Barium	200		75.1	75.8	0.9	None
Beryllium	5		1.0 U	1.0 U	NC	None
Cadmium	5		3.0 U	3.0 U	NC	None
Calcium	5000		53500	51500	3.8	None
Chromium	10		8.0	13.3	49.8	None
Cobalt	60		4.0 U	6.1	200	None
Copper	25		16.1	14.8	8.4	None
Iron	100		9860	12400	22.8	None
Lead	3		8.8	8.4	4.7	None
Magnesium	5000		5980	6300	5.2	None
Manganese	15		4500	4340	3.6	None
Mercury	0.2		0.20 U	0.20 U	NC	None
Nickel	40		22.0	22.3	1.4	None
Potassium	5000		7400	7450	0.7	None
Selenium	5		4.0 U	4.0 U	NC	None
Silver	10		5.0 U	5.0 U	NC	None
Sodium	5000		115000	109000	5.4	None
Thallium	10		15.0 U	3.0 U	NC	None
Vanadium	50		12.4	14.2	13.5	None
Zinc	20		31.7	30.5	3.9	None
Cyanide	10		10.0 U	10.0 U	NC	None

Field Duplicate Actions should be applied to all other samples of the same matrix type.

### ACTIONS:

1. Estimate (J) positive results for elements which have an RPD >30% for waters and >50% for soils.
2. If sample results are less than 5x the CRDL, estimate (J) positive results and (UJ) non-detected results for elements whose absolute difference is >2xCRDL (4xCRDL for soils). If both samples are non-detected, the RPD is not calculated (NC).

**IX. LABORATORY CONTROL SAMPLE**

**1. Aqueous LCS**

List any LCS recoveries not within the 80-120% criteria and the samples affected.

<u>DATE</u>	<u>ELEMENT</u>	<u>%R</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
None				

**2. Solid LCS**

List any analytes that were not within the control windows set by the EPA for the solid LCS sample. The 80-120% criteria is not used to evaluate solid LCS results.

<u>ELEMENT</u>	<u>LCS CONC.</u>	<u>CONTROL WINDOWS</u>	<u>ACTION</u>	<u>SAMPLES AFFECTED</u>
None				

**ACTIONS:**

**Aqueous LCS**

	<u>Percent Recovery</u>		
	<u>&lt;50%</u>	<u>51%-79%</u>	<u>&gt;120%</u>
Positive sample results	R	J	J
Non-detected results	R	UJ	A

**Solid LCS**

	<u>&lt;EPA Control Windows</u>	<u>&gt;EPA Control Windows</u>
Positive sample results	J	J
Non-detected results	UJ	A

**3. Frequency Criteria**

Was an LCS analyzed for every matrix, every digestion batch, and every 20 samples?

Yes

## X A. FURNACE ATOMIC ABSORPTION ANALYSIS

### 1. Duplicate Precision

X Duplicate injections and one-point analytical spikes were performed for all samples: duplicate injections agreed within  $\pm 20\%$ .

\_\_\_\_\_ Duplicate injections and/or spikes were not performed for the following samples/elements: \_\_\_\_\_

\_\_\_\_\_ Duplicate injections did not agree within  $\pm 20\%$  for samples/elements: \_\_\_\_\_

### 2. Post Digestion Spike Recoveries

\_\_\_\_\_ Spike recoveries met the 85-115% recovery criteria for all samples.

X Spike recoveries did not meet the 85-115% criteria but did not require MSA for the following samples/elements: Se (MAT368, MAT369, MAT370, MAT371)  
Tl (MAT367, MAT368, MAT370, MAT371)

X MSA was used to quantitate analytical results when contractually required.

X Correlation coefficients  $\geq 0.995$ , accept results.

\_\_\_\_\_ Correlation coefficients  $< 0.995$  for sample numbers/elements: \_\_\_\_\_

### ACTIONS:

1. Estimate (J) positive results if duplicate injections are outside  $\pm 20\%$  RSD or CV.
2. If the sample absorbance is  $< 50\%$  of post digestion spike absorbance, the following actions should be applied:

	<u>Percent Recovery</u>		
	<u><math>&lt; 10\%</math></u>	<u>11%-84%</u>	<u><math>&gt; 115\%</math></u>
Positive sample results	J or R	J	J
Non-detected results	R	UJ	A

3. Estimate (J) sample results if MSA was required and not performed.
4. Estimate (J) sample results if correlation coefficient was  $< 0.995$ .

# **XI. INDUCTIVELY COUPLED PLASMA (ICP) SERIAL DILUTION ANALYSIS**

Serial Dilutions were performed for each matrix and results of the diluted sample analysis agreed within 10% of the original undiluted analysis.

Serial Dilutions were not performed for the following:\_\_\_\_\_

X Serial Dilutions were performed, but analytical results did not agree within 10% for analyte concentrations greater than 50x the IDL before dilution.

Report all results below that do not meet the required laboratory criteria for ICP serial dilution analysis.

MATRIX: Soil

<u>ELEMENT</u>	<u>IDL</u>	<u>50xIDL</u>	<u>SAMPLE RESULT</u>	<u>SERIAL DILUTION</u>	<u>%D</u>	<u>ACTION</u>
Aluminum						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium	3.0	150	162.34	137.15	15.5	J/A
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Nickel						
Potassium						
Silver						
Sodium						
Vanadium						
Zinc						

Actions apply to all samples of the same matrix.

## **ACTIONS:**

1. Estimate (J) positive results if %D >15.

## XII. DETECTION LIMIT RESULTS

### 1. Instrument Detection Limits

  X   Instrument Detection Limit results were present and found to be less than the Contract Required Detection Limits.

  X   Instrument Detection Limits were not included in the data package on Form X.

       Instrument Detection Limits were present, but the criteria was not met for the following elements: \_\_\_\_\_

### 2. Reporting Requirements

       Were sample results on Form I reported down to the IDL not the CRDL for all analytes? Yes

  N/A   Were sample results that were analyzed by ICP for Se, Tl, As, or Pb at least 5x IDL?

       Were sample weights, volumes, and dilutions taken into account when reporting detection limits on Form I? Yes

If no,  
the reported results may be inaccurate. Make the necessary changes on the data summary tables and request that the laboratory resubmit the corrected data.

No IDL for CN.

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### XIII. SAMPLE QUANTITATION

  X   Sample results fall within the linear range for ICP and within the calibrated range for all other parameters.

       Sample results were beyond the linear range/calibration range of the instrument for the following samples/elements: \_\_\_\_\_

In the space below, please show a minimum of one sample calculation per method:

#### ICP:

All samples within this SDG were water samples.

#### FURNACE:

All samples within this SDG were water samples.

#### MERCURY:

All samples within this SDG were water samples.

#### CYANIDE:

All samples within this SDG were water samples.

For soil samples, the following equation may be necessary to convert raw data values (usually reported in ug/L) to actual sample concentrations (mg/kg):

The lab is required to use 1 gram sample (wet weight) to 200 ml.

$$\text{Wet weight concentration} = \frac{\text{digest conc. in ug}}{\text{L}} \times \frac{200 \text{ ml}}{1 \text{ gm}} \times \frac{1 \text{ L}}{1000 \text{ ml}} \times \frac{1000 \text{ gm}}{1 \text{ kg}} \times \frac{1 \text{ mg}}{1000 \text{ ug}} = \frac{\text{mg}}{\text{kg}}$$

In addition, the sample results are converted to dry weight using the percent solids calculations:

$$\frac{\text{Wet weight conc.}}{\% \text{ solids}} \times 100 = \text{final concentration, dry weight (mg/kg)}$$

CLP INORGANIC ANALYSIS  
CASE NO. 16259 SDG NO. MAT367  
ANALYTICAL RESULTS

Sample Location		Ciba Gelgy	Ciba Gelgy	Ciba Gelgy	Ciba Gelgy	Ciba Gelgy	Ciba Gelgy		
Sample Number		MW-145	MW-145	MW-115	MW-155	MW-185			
Traffic Report Number		MAT367	MAT368	MAT369	MAT370	MAT371	MAT372		
Remarks			Dup. of MAT367				Field Blank		
Sampling Date		04/18/91	04/18/91	04/18/91	04/18/91	04/18/91	04/18/91		
Inorganic Elements		ug/L	ug/L	ug/L	ug/L	ug/L	ug/L		
Aluminum P		3680 J	5340 J	5460 J	5070 J	10600 J	20.5 J		
Antimony P									
Arsenic F		19.7	13.3	32.3	36.2	16.1			
Barium P		75.1	75.8		132	117			
Beryllium P				1.2					
Cadmium P									
Calcium P		53500	51500	25000	52700	31500			
Chromium P		8.0 J	13.3 J	23.9 J	34.9 J	162 J			
Cobalt P									
Copper P							9.4 J		
Iron P		9860	12400	32500	31000	17300			
Lead F		8.8	8.4	23.2	7.5	8.1	1.1		
Magnesium P		5980	6300	4070	6560	5730			
Manganese P		4500	4340	352	1930	1210			
Mercury V									
Nickel P		22.0	22.3	13.8	70.0	275			
Potassium P		7400	7450	3530	13800	6930			
Selenium F									
Silver P									
Sodium P		115000	109000	11300	27100	21300	128		
Thallium F									
Vanadium P		12.4	14.2	12.8	11.6	21.9			
Zinc P				2420	99.0	61.9	10.3 J		
Cyanide C									

## Analytical Method

F Furnace  
P ICP/Flame AA  
V Cold Vapor  
C Colorimetric

A blank space indicates the element was not detected.

J Quantitation is approximate due to limitations identified in the quality control review.

R Value is rejected.

NA Not Analyzed

Sample Detection Limits for the elements listed above are reported in Table 8.



CLP INORGANIC ANALYSIS  
CASE NO. 16259 SDG NO. MAT367  
SAMPLE DETECTION LIMITS

Sample Location			Ciba Gelgy	Ciba Gelgy	Ciba Gelgy	Ciba Gelgy	Ciba Gelgy	Ciba Gelgy		
Sample Number			MW-145	MW-145	MW-115	MW-155	MW-185			
Traffic Report Number			MAT367	MAT368	MAT369	MAT370	MAT371	MAT372		
Remarks				Dup. of MAT367				Field Blank		
Sampling Date			04/18/91	04/18/91	04/18/91	04/18/91	04/18/91	04/18/91		
Percent Solids			0.0	0.0	0.0	0.0	0.0	0.0		
Inorganic Elements			Instrument Detection Limits (ug/L)	ug/L	ug/L	ug/L	ug/L	ug/L		
Aluminum	P	14.0	14.0	14.0	14.0	14.0	14.0	14.0		
Antimony	P	17.0	17.0	17.0	17.0	17.0	17.0	17.0		
Arsenic	F	2.0	2.0	2.0	2.0	2.0	2.0	2.0		
Barium	P	2.0	2.0	2.0	36.0	2.0	2.0	2.0		
Beryllium	P	1.0	1.0	1.0	1.0	1.0	1.0	1.0		
Cadmium	P	3.0	3.0	3.0	3.0	3.0	3.0	3.0		
Calcium	P	15.0	15.0	15.0	15.0	15.0	15.0	15.0		
Chromium	P	3.0	3.0	3.0	3.0	3.0	3.0	3.0		
Cobalt	P	4.0	4.0	6.1 UJ	4.0	5.9 UJ	11.7 UJ	4.0		
Copper	P	4.0	16.1 UJ	14.8 UJ	41.3	11.7 UJ	25.3	4.0		
Iron	P	8.0	8.0	8.0	8.0	8.0	8.0	8.0		
Lead	F	1.0	1.0	1.0	1.0	1.0	1.0	1.0		
Magnesium	P	29.0	29.0	29.0	29.0	29.0	29.0	29.0		
Manganese	P	1.0	1.0	1.0	1.0	1.0	1.0	1.0		
Mercury	V	0.2	0.2	0.2	0.2	0.2	0.2	0.2		
Nickel	P	5.0	5.0	5.0	5.0	5.0	5.0	5.0		
Potassium	P	72.0	72.0	72.0	72.0	72.0	72.0	72.0		
Selenium	F	4.0	4.0 UJ	4.0 UJ	4.0 UJ	4.0 UJ	20.0 UJ	4.0 UJ		
Silver	P	5.0	5.0	5.0	5.0	5.0	5.0	5.0		
Sodium	P	24.0	24.0	24.0	24.0	24.0	24.0	24.0		
Thallium	F	3.0	15.0 UJ	3.0 UJ	3.0	3.0 UJ	3.0 UJ	3.0		
Vanadium	P	3.0	3.0	3.0	3.0	3.0	3.0	3.0		
Zinc	P	7.0	31.7	30.5	7.0	7.0	7.0	7.0		
Cyanide	C	10.0	10.0	10.0	10.0	10.0	10.0	10.0		

## Analytical Method

F Furnace AA

P ICP/Flame AA

V Cold Vapor

C Colorimetric

Sample's wet weight (gms) digested:

for Hg analysis								
for ICP analysis								
for furnace AA analysis								
for Cyanide analysis								

Volumes used in preparing sample for analysis:

for Hg analysis	100 mls
for ICP and AA analysis	200 mls
for Cyanide analysis	250 mls

UJ Value is undetected and the quantitation is approximate due to limitations identified in the quality control review.

R Value is rejected.